



Europäisches
Patentamt

European
Patent Office

Office européen
des brevets

Bescheinigung

Certificate

Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

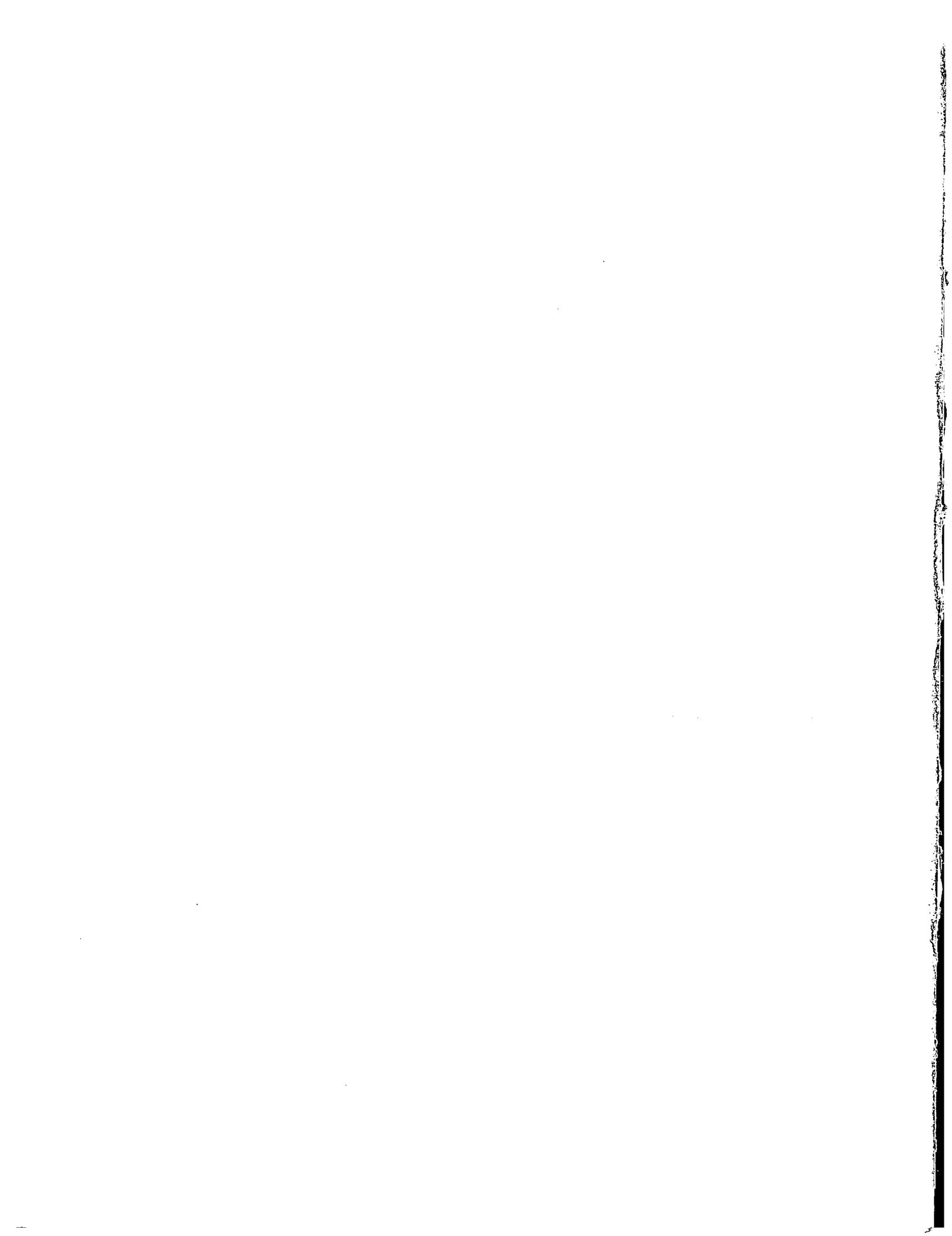
02023662.6

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

R C van Dijk





Anmeldung Nr:
Application no.: 02023662.6
Demande no:

Anmeldetag:
Date of filing: 22.10.02
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

Bayer Aktiengesellschaft

51368 Leverkusen
ALLEMAGNE

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

Process for the purification of toluenediisocyanate incorporating a dividing-wall distillation column for the final purification

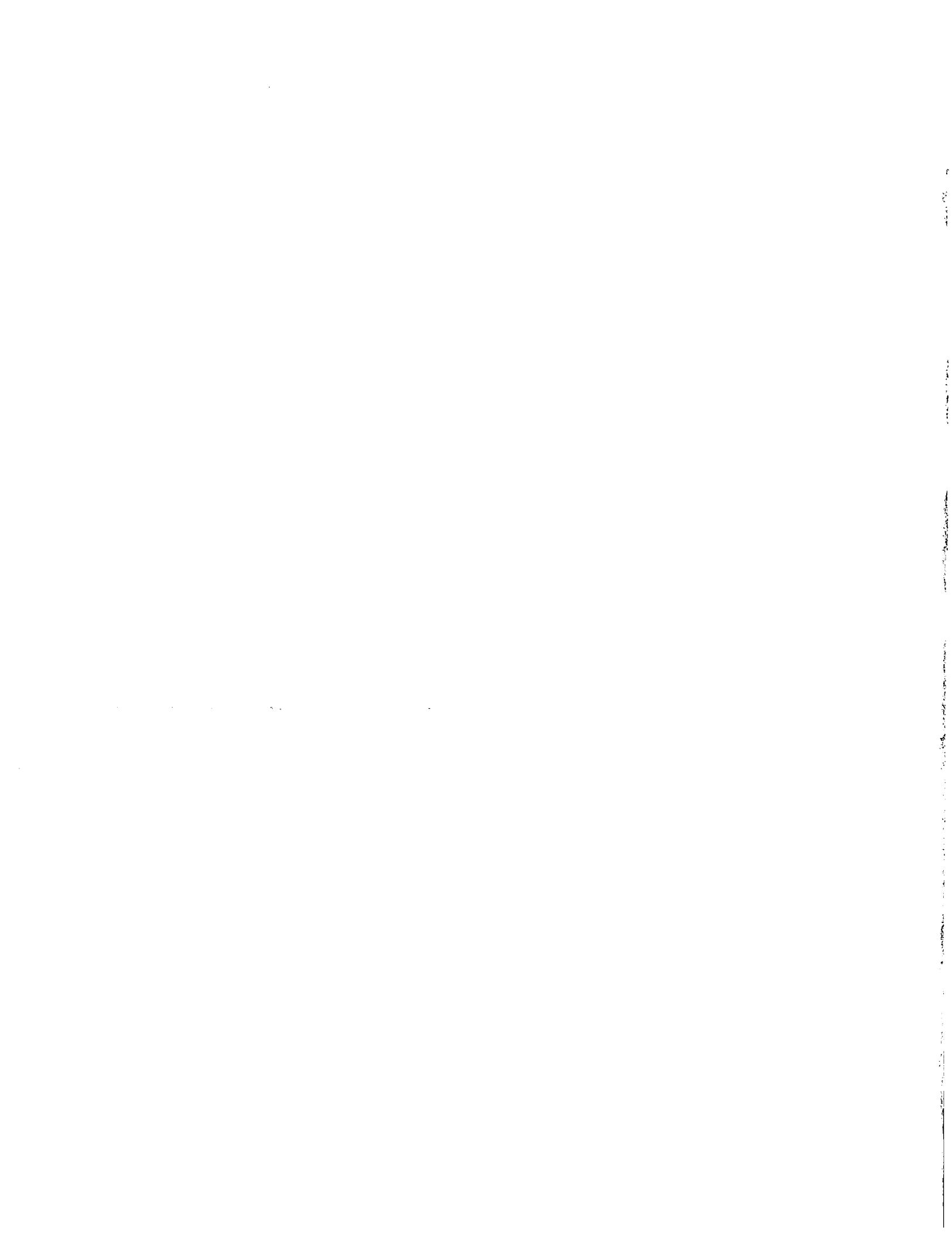
In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)
revendiquée(s)
Staat/Tag/Aktenzeichen/State>Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/
Classification internationale des brevets:

C07C263/00

Am Anmeldestag benannte Vertragstaaten/Contracting states designated at date of
filing/Etats contractants désignés lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR IE IT LI LU MC NL PT SE SK TR



Process for the purification of toluenediisocyanate incorporating a dividing-wall distillation column for the final purification

The present invention relates to an improvement of a toluenediisocyanate (TDI) recovery and purification process which uses a dividing wall column for the final purification of the TDI product. The process of the present invention benefits from the ability to achieve a higher TDI purity.

The present invention relates to a process wherein toluediamine is reacted with phosgene in the presence of a solvent solution in the liquid phase or wherein toluediamine is reacted with phosgene directly in the gas phase with a solvent used in the quench cooling of said reaction; excess phosgene is then partially or completely removed from the resulting reaction mixture and the dephosgenated crude distillation feed fed to a fractionation process wherein the solvent and optionally the residue is removed. The subsequent crude TDI feed is fed to a dividing-wall distillation column wherein four fractions are recovered

- 1) a vapor phase low-boiler and solvent enriched product from which the condensable species are preferably recovered and returned to the dephosgenation, residue removal, or solvent removal process.
- 2) a low-boiler enriched product which is then preferably returned to the dephosgenation, residue removal, or solvent removal process or recovered as a separate product stream.
- 3) a high-boiler enriched bottoms product which is preferably sent to a residue removal system for the further recovery of volatiles.
- 4) an isocyanate product stream.

The field of art to which this invention pertains is a process for the purification of toluenediisocyanate (TDI) mixtures. TDI mixtures are generally produced by reacting toluene with nitric acid to yield dinitrotoluene (DNT), hydrogenating the resultant dinitrotoluene (DNT) to yield toluediamine (TDA) and reacting the toluediamine

(TDA) with phosgene to give toluenediisocyanate (TDI). Toluenediisocyanate (TDI) is a commercial available material particularly useful in the preparation of polyurethanes, polyurea and polyisocyanurate polymers, especially foamed polymers.

5 DE-A1-3736988 teaches that organic mono- or poly-isocyanates are continuously prepared by reacting the corresponding mono- or poly-amine dissolved in an inert organic solvent with phosgene also dissolved in an inert organic solvent at a temperature under 150°C. The amine and phosgene solutions are combined and allowed to pass through one or more reaction columns connected below to above in
10 series and having at least 10 chambers in total separated from each other by perforated plates, the holes of which preferably have a max diameter of 20 mm.

15 EP-A1-570799 teaches that production of aromatic diisocyanates is effected by reaction of diamines and phosgene. The phosgene and diamine are at above the boiling temperature of the diamine and the reaction has an average contact time of 0.5-5 seconds. The mixture is continuously passed through a cylindrical reaction space at 200 – 600°C to complete the reaction with avoidance of back mixing. The gas mixture is then cooled to condense the diisocyanates, with the temperature being maintained above the decomposition temperature of carbamic acid chlorides
20 corresponding to the diamines used. Uncondensed diisocyanate is washed out of the gas mixture with an inert solvent, and the inert solvent is recovered by distillation.

25 The *Polyurethane Handbook* (Oertel, G. (Editor), Polyurethane Handbook, Munich, Germany: Hanser Publishers, 1985, pp 62-73) gives a description of a state of the art for the phosgenation and distillation process for the production of toluenediisocyanate. In the distillation process, the solvent is completely removed from the crude TDI mixture as the top product from a solvent column, with this solvent being returned to the phosgenation or to the excess phosgene recovery. The remaining crude isocyanate bottoms stream from the solvent column is sent to a pre-flasher where two products are achieved: a isocyanate rich overhead product and a residue-enriched bottoms stream which is fed to the residue removal. In the residue
30

removal, the volatiles are then removed from this residue-enriched stream and condensed. The condensed volatiles from residue removal together with the condensed overhead stream from the pre-evaporation are then combined and fed to an isocyanate column. In the isocyanate column, the product isocyanate is recovered as a top stream while a high-boiler enriched bottoms stream is returned to the pre-evaporation step. This process is limited by the fact that the complete solvent removal is performed in one solvent column. While it is known that TDI yields are negatively affected by higher temperatures, complete solvent removal necessitates operating under relatively low pressures to achieve sump temperatures low enough to prevent a loss of yield, thus necessitating a large column. Moreover, the long residence-time of isocyanate together with residue in heating zones can lead to a higher rate of residue formation. Finally, condensation of the overhead stream from the pre-evaporation before feeding to the isocyanate column is energy inefficient.

- 15 In *Industrielle Aromatenchemie* (Franck H.-G. and Stadelhofer J., *Industrielle Aromatenchemie*. Berlin, Germany: Springer Verlag, 1987, p 253) a second state-of-the-art process is described . In the described process, the crude TDI-solvent mixture is fed to a two-step pre-evaporation step resulting in a low-boiling overhead vapor product and solvent-free residue-enriched bottoms product which is fed to the residue removal. In the residue removal process, the volatiles are then removed from this residue-enriched stream and condensed. The overhead product from the pre-evaporation is fed to a solvent column. In the solvent column the solvent is completely removed as the top product, with the solvent being returned to the phosgenation or to the excess phosgene recovery. The remaining crude isocyanate bottoms stream from the solvent column is fed along with the condensed volatiles from residue removal to an isocyanate column. In the isocyanate column, the product isocyanate is recovered as a top stream while a high-boiler (polymeric isocyanate and hydrolyzable chloride compounds (HCC), and other non-volatiles) enriched bottoms stream is returned to the pre-evaporation step. This process is also limited by the fact that the complete solvent removal must be performed in one solvent column. As in the process described in the *Polyurethane Handbook*,

complete solvent removal necessitates operating under relatively low pressures to achieve sump temperatures low enough to prevent a loss of yield, resulting in a large solvent column. However, this process, in comparison with the former process achieves a reduced residence-time of isocyanate together with residue in heating
5 zones possibly leading to a lower rate of residue formation. Moreover, because there is no needless condensation of a vapor feed to the isocyanate column, this process will be more energy efficient.

From Chem System's *PERP Report for TDI/MDI* (Chem Systems, Process
10 Evaluation Research Planning TDI/MDI 98/99S8. Tarrytown, NY, USA: Chem Systems, 1999, pp 27-32) for TDI/MDI it can be learned, that the fractionation of a crude TDI distillation feed product can be completed in the following manner. Normally, the liquid product from the dephosgenation stage is sent to a pre-evaporator which produces a residue-rich liquid-phase as a bottom product and a
15 vapor-phase product containing mainly solvent and isocyanate as an overhead product. The bottom product from the pre-evaporation is sent to a process for the removal of volatile compounds from the reaction residues (residue removal). The volatile components removed in the residue removal stage as well as the vapor-phase product from the pre-evaporator are sent to a solvent column, where an initial separation of the isocyanate from solvent is completed as well as the removal of any
20 remaining phosgene. The resulting products are a phosgene-enriched top product, a relatively pure solvent stream as an intermediate product and an isocyanate-enriched bottoms product. The phosgene stream is then returned to the dephosgenation process or to the excess phosgene recovery process. The solvent product is then used
25 in the phosgenation section as well as in the excess phosgene recovery. The bottoms isocyanate-rich product is then sent to a second solvent removal column where the remainder of the solvent is removed. The top solvent product from this steps, when relatively pure, can be used in phosgenation or excess phosgene recovery or can be returned to the primary solvent removal step. The final solvent-free bottoms isocyanate product is sent to an isocyanate column, resulting in an isocyanate top product and a residue and hydrolyzable chloride compound (HCC) enriched-bottom
30

stream which is returned to the pre-evaporation or to the residue-removal stages. This process like the process described in *Industrielle Aromatenchemie*, in comparison with the process described in the *Polyurethane Handbook* achieves a reduced residence-time of isocyanate together with residue in heating zones possibly leading to a lower rate of residue formation. Additionally, like the process described in *Industrielle Aromatenchemie*, because there is no needless condensation of a vapor feed to the isocyanate column, this process will be more energy efficient than the process disclosed in the Polyurethane Handbook. It holds the additional advantage, that the solvent removal is completed in two-steps. By taking advantage that the solvent has a lower boiling point than the isocyanate, the majority of the solvent can be removed under higher pressure, therefore, reducing the necessary investment cost for the solvent removal. Additionally, the use of two solvent removal steps adds to the flexibility of operation. However, the presence of a third column adds more complexity to the process.

15

In fractionation, it is sometimes desirable to separate a multi-component feed stream into a number of streams containing, various fractions of desirable components in the product streams. For the case of one feed stream and two product streams, the separation can be accomplished by distillate and bottoms product draw. Further separation can be accomplished by repeating the two-product stream process to either the distillate or the bottoms streams. However, the introduction of additional columns will require a corresponding number of reboilers and condensers. That requirement, in turn, requires additional operating costs as the condensing and the reboiling process is being repeated. Numerous references can be found in prior art documenting efforts to lower both capital and operating costs in the separation of several fractions from a multi-component feed stream. The benchmark of the lowest energy consumption has been set by the old and well-known PETLYUK system (Agrawal, R and Fidkowski, Z, Are Thermally Coupled Distillation Columns Always Thermodynamically More Efficient for Ternary Distillations?, *Industrial & Engineering Chemistry Research*, 1998, 37, pp 3444-3454). In this configuration, a prefractionation column separates the feed into two streams using a split vapor

stream from the main column's stripping section and a split liquid stream from the main column's rectifying section. The resulting vapor and liquid streams exiting from the prefractionation column is richer in light and heavy components respectively. These two semi-processed streams are then fed back to the main column. This configuration provides an advantage allowing the main fractionation column to enhance the purity of the side stream draw. In turn, the main fractionation column also provides the stripping section and the rectifying section with better quality feeds. The combined effect is a very efficient use of vapor / liquid traffic to yield three product streams.

10

US-A-2,471,134 teaches an improvement of the Petyluk process with a proposition to combine the prefractionation and main columns into one fractionation unit by erecting a partition along the center part of a column. The column is equipped with one overhead condenser and one bottom reboiler.

15

The dividing-wall distillation column according to US-A-2,471,134 is a vertical column fractionating tower, equipped with reboiler and condenser, which is divided into four distinct column sections by the use of a center partition in the intermediate part of the column. These sections are a common bottom (stripping) and top (rectifying) sections, and the prefractionation and main fractionation sections in the intermediate part of the column separated by a dividing-wall. The multi-component mixture is fed to the prefractionation section, the overhead product is taken from the common rectifying section, a bottoms product is taken from the common stripping section, and the intermediate product stream is taken as a side-product from the main fractionation section. One significant advantage of the use of a dividing-wall distillation column is the fact that the sidedraw product can be obtained from the dividing wall distillation column with a lower-concentrations of low-boiling impurites than that of a side product which is obtained from a simple sidedraw product column.

20
25
30

This dividing-wall distillation column is effective in overcoming the hydraulic limitations in the PETLYUK system. At the same time, it reduces capital costs by having only one common shell. The dividing-wall distillation column disclosed in US-A-2,471,134 has found applications in several processes.

5

In the present invention, the use of the dividing-wall distillation column for the final purification of the TDI product in the TDI distillation process allows for a surprising improvement in TDI purity by the reduction of the amount of low-boiling impurities with reduced energy requirements and investment costs as compared to a standard sidedraw column to achieve the same product quality. Examples of these impurities include solvents, solvent impurities, aromatic monoisoxyanates, chlorinated aromatic monoisoxyanates, etc.

10

The invention is directed to a process for the purification of toluenediisocyanate from a crude distillation feed comprising less than 2% by weight of phosgene by

15

a) fractionating the crude distillation feed comprising less than 2% by weight of phosgene to remove the solvent and optionally the reaction residues to produce a crude toluylenediisocyanate feed comprising less than 20% by weight of solvent and

20

b) separating the crude toluylenediisocyanate feed comprising less than 20% by weight of solvent in a dividing-wall distillation column into four product fractions P1 – P4, whereby

25

P1 is a vapor phase low-boiler and solvent-enriched gas stream,

P2 is a low-boiler and solvent-enriched product,

P3 is a high boiler enriched bottoms product comprising toluenediisocyanate and

P4 is a toluenediisocyanate product stream lean in low-boilers, high-boilers and reaction residues.

30

The invention is also directed to a process for the production of toluenediisocyanate comprising the steps

- a) reacting toluene diamine with phosgene resulting in a crude distillation feed,
 - 5 b) separating the phosgene from the crude distillation feed from step a) if the crude distillation feed from step a) comprises 2 % by weight or more of phosgene resulting in a crude distillation feed comprising less than 2% by weight of phosgene,
 - 10 c) fractionating the crude distillation feed comprising less than 2% by weight of phosgene to remove the solvent and optionally the reaction residues to produce a crude toluylenediisocyanate feed comprising less than 20% by weight of solvent and
 - 15 d) separating the crude toluylenediisocyanate feed comprising less than 20% by weight of solvent in a dividing-wall distillation column into four product fractions P1 – P4, whereby
- P1 is a vapor phase low-boiler and solvent-enriched gas stream,
P2 is a low-boiler and solvent-enriched product,
P3 is a high boiler enriched bottoms product comprising toluenediisocyanate and
20 P4 is a toluenediisocyanate product stream lean in low-boilers, high-boilers and reaction residues.

The phosgenation is performed according to the state of the art. Toluene diamine is reacted with phosgene in the presence of a solvent solution in the liquid phase or 25 with phosgene directly in the gas phase with a solvent used in the quench cooling of said reaction. The resulting reaction mixture preferably has a composition of 5 – 40% by weight toluenediisocyanate, 1-2 % by weight hydrogen chloride, 1-5 % by weight phosgene, 0.1 - 2% by weight high-boilers (polymeric isocyanates, hydrolyzable chloride compounds (HCC)), and the rest being solvent. In this case 30 hydrolyzable chloride compounds are defined as compounds in which the available

chlorine is "loosely" bound. Illustrative of these compounds are the following species : $\text{ClCH}_2\text{C}_6\text{H}_3(\text{NCO})_2$ and $(\text{CH}_3\text{NCOCl})\text{CH}_3\text{C}_6\text{H}_3(\text{NCO})$

5 Content of hydrolyzable chloride compounds are generally determined by reacting the available chorine in the sample with a hot water-alcohol solution resulting in HCl and a subsequent titration to determine the hydrolyzable chlorine concentration. This value is generally reported as weight fraction hydrolyzable chlorine" (HC).

10 Chlorinated aromatic hydrocarbons are species in which the chlorine is "tightly" bound. Illustrative of such componds are the common solvents o-dichlorobenzene, and chlorobenzene, and related compounds .

15 After the reaction the resulting reaction mixture is fed to a separation step if the reaction mixture (crude distillation feed) comprises 2% by weight or more of phosgene. In this separation step, the excess phosgene is at least partly removed resulting in crude distillation feed comprising less than 2% by weight of phosgene. The separation of the phosgene can be performed using many different methods or combinations thereof. Examples of these methods are simple vapor/liquid flash separation, with or without the increase of temperature or a decrease in pressure, gas 20 stripping, distillation, etc.

25 The resulting crude distillation feed comprising less than 2% by weight of phosgene is then fed to a distillation column or system of distillation columns wherein the solvent and optionally the reaction residues is removed from the crude distillation feed comprising less than 2% by weight of phosgene to produce a crude toluylene-diisocyanat feed comprising less than 20% by weight of solvent. The distillation column(s) used for the removal of the solvent and optionally the reaction residues may be a conventional distillation column or a dividing wall distillation column. Preferably, a conventional distillation column according to the state of the art is used.

- 10 -

The resulting crude toluylenediisocyanat feed comprising less than 20% by weight of solvent is then fed to a dividing wall distillation column and separated into four product fractions P1 – P4.

5 Product Fraction P1 is a vapor-phase low-boiler and solvent-enriched product comprising 20-99% by weight of condensable species (i.e. solvent, low-boilers and TDI) with the rest being noncondensable gases, i.e. air, hydrogen chloride, etc. The condensable fraction of this product may comprise solvent, low-boilers and TDI. The condensable species are preferably recovered and returned to the dephosgenation, 10 residue removal, or solvent removal process.

15 Product Fraction P2 is a low-boiler and solvent-enriched product which is then preferably returned to the dephosgenation, residue removal, or solvent removal process or recovered as a separate product stream. The product fraction P2 may comprise solvent, low boilers and TDI.

20 Product Fraction P3 is a high-boiler enriched bottoms product which is preferably sent to a residue removal system for the further recovery of volatiles. The fraction P3 preferably comprises 0.5-15% by weight high-boilers (polymeric isocyanates, hydrolyzable chloride compounds (HCC), and other non-volatiles), the rest mainly being toluenediisocyanate.

25 Product Fraction P4 is an isocyanate product stream. The fraction P4 preferably comprises less than 200 ppm by weight of solvent and / or chlorinated aromatic hydrocarbons (in total), less than 100 ppm by weight hydrolyzable chlorine (HC) , less than 40 ppm by weight acidity, with a toluenediisocyanate concentration of at least 99.5% by weight.

30 The fractionation process including a dividing-wall distillation column may be successfully utilized for the purification of a partially to fully dephosgenated TDI reaction product resulting from the reaction of toluene diamine with phosgene in the

presence of a solvent solution or from this reaction in the gas phase with a solvent used in the quench cooling after the reaction. The resulting distillation feed contains phosgene and other low-boiling components, solvent, toluene diisocyanate, hydrolyzable chloride compounds, and high-boiling residues. This stream is in turn fractionated to achieve the removal of the solvent and optionally the reaction residue to attain crude TDI which is then fed to a dividing-wall TDI purification column. The four products from the dividing-wall column are a low-boiler and inert gas-enriched product, from which the condensable species are preferably recovered and returned to the dephosgenation, residue removal, or solvent removal process, a low-boiler enriched liquid product which is then preferably returned to the dephosgenation, residue removal, or solvent removal process or recovered as a separate product stream, a high-boiler enriched bottoms product which is preferably sent to a residue removal system for the further recovery of volatiles and an isocyanate product stream. The solvent to be used can be any suitable solvent, preferably o-dichlorobenzene, p-dichlorobenzene, chlorobenzene, toluene, benzene, nitrobenzene, anisole, xylene, or any mixture thereof. Depending on reaction conditions different concentrations of TDI in the crude distillation feed can be obtained.

The final purification of the crude toluylenediisocyanate feed comprising less than 20% by weight of solvent is performed in a dividing-wall distillation column according to Fig. 1. This dividing-wall distillation column is at least equipped with one reboiler and one condenser. The reboiler can be any of the standard types commonly found in the chemical industry, including in part falling-film evaporators, forced circulation evaporators, pool boiling (kettle) evaporators, natural circulation evaporators, etc. The condenser can be any of the types in common use in the chemical industry including co-current and countercurrent (knockback condensers). The column can be equipped with any mass transfer internals that are in common use in the chemical industry. These include, in part, sieve trays, valve trays, fixed valve trays, as well as structured or random distillation packings.

The invention is described in more detail in the following with reference to the accompanying drawing, wherein

5 Figure 1 shows a schematic of the diving wall distillation column which is used in the process for the purification of mixtures of TDI,

Figure 1 shows a dividing-wall distillation column 1 which is equipped with a reboiler 2, a condenser 3 a dividing-wall 4 and mass transfer internals 5.

10 The diving wall distillation column 1 is divided into four distinct operating zones, a prefractionation zone where the feed is introduced, a stripping zone with the high-boiler product P3, a main fractionation zone with the isocyanate product P4, and a rectifying zone with a vapor phase low-boiler product P1, and a liquid-phase low-boiler and solvent-enriched product P2. The prefractionation and the main
15 fractionation zones lay side by side in the diving wall distillation column 1 with a dividing-wall 4 separating the two zones.

Prefractionation Zone

20 The crude distillation feed A is fed to the prefractionation zone, wherein it is separated into two streams, a residue and a hydrolyzable chloride compound (HCC) - enriched liquid TDI stream B and a low-boiler enriched vapor stream C. This separation is effected by two streams, one liquid D and one vapor E. The liquid stream D, containing both low-boilers and TDI, enters the prefractionation zone from the rectifying zone. The vapor stream E, containing TDI and HCC's enters the prefractionation zone from the stripping zone.
25

Stripping Zone

30 The liquid product B from the prefractionation zone as well as the TDI and HCC-containing liquid product F from the main fractionation zone enters the upper

section of the stripping zone. Vapor G generated from the reboiler 2 in stream R causes the separation of intermediate component from the heavy component. The resulting residue-enriched liquid containing highboiler is routed away as the bottoms product stream P3. The column is designed for an operating pressure so that the temperature achieved in the reboiler will preferably be in the range of from 140-190°C. The TDI-enriched vapor streams E and H are fed to the prefractionation zone and the main fractionation zone respectively. The distribution of the vapor flow to the prefractionation zone and main fractionation zone is effected by the inherent pressure drop in the respective column section.

10

Rectifying Zone

The low-boiler enriched vapor products C from the prefractionation zone and I from the main fractionation zone, both containing intermediate as well as low-boiling components enters the rectifying zone at the lower section. The vapor product J from the rectifying zone is fed to a condenser 3, and then a portion of the condensate product generated from the condenser is returned as reflux K to the top of the rectifying zone causing the separation of light component from the intermediate component. The remaining fraction of the condenser liquid product is routed away as the low-boiler and solvent-enriched liquid product stream P2. The uncondensed vapor product from the condenser is the low-boiler product stream P1. Internal reflux within the column generates a liquid stream. This liquid stream, containing mainly low-boilers and TDI, is divided into streams L and D which are routed to the main fractionation zone and to the prefractionation zone, respectively. The proportional distribution of these liquid streams are controlled to achieve the required product quality. Optionally the product, P2 can be taken as a sidedraw product from any stage in the rectifying zone.

15

20

25

Main Fractionation Zone

A TDI-enriched vapor product stream H from the stripping zone enters the main fractionation zone from the bottom. A portion of the liquid product L from the
5 rectifying zone enters the main fractionation zone from the top. The resulting fractionation generates three products: a vapor feed I to the rectifying zone, and a liquid feed F to the stripping zone, and a side draw product P4 that contains the desired quality isocyanate product. Optionally, the product P2 can be taken as a
10 sidedraw product from any stage above the product removal stage for P4 in the main fractionation zone.

Claims:

1. Process for the purification of toluenediisocyanate from a crude distillation feed comprising less than 2% by weight of phosgene by

5 a) fractionating the crude distillation feed comprising less than 2% by weight of phosgene to remove the solvent and optionally the reaction residues to produce a crude toluylenediisocyanate feed comprising less than 20% by weight of solvent and

10 b) separating the crude toluylenediisocyanat feed comprising less than 20% by weight of solvent in a dividing-wall distillation column into four product fractions P1 – P4, whereby

P1 is a vapor phase low-boiler and solvent-enriched gas stream,

P2 is a low-boiler and solvent-enriched product,

15 P3 is a high boiler enriched bottoms product comprising toluenediisocyanat and

P4 is a toluenediisocyanate product stream lean in low-boilers, high-boilers and reaction residues.

2. Process for the production of toluenediisocyanate comprising the steps

20 a) reacting toluene diamine with phosgene resulting in a crude distillation feed,

b) separating the phosgene from the crude distillation feed from step a) if the crude distillation feed from step a) comprises 2 % by weight or more of phosgene resulting in a crude distillation feed comprising less than 2% by weight of phosgene,

25 c) fractionating the crude distillation feed comprising less than 2% by weight of phosgene to remove the solvent and optionally the reaction residues to produce a crude toluylenediisocyanat feed comprising less than 20% by weight of solvent and

d) separating the crude toluylenediisocyanat feed comprising less than 20% by weight of solvent in a dividing-wall distillation column into four product fractions P1 – P4, whereby

P1 is a vapor phase low-boiler and solvent-enriched gas stream,

5

P2 is a low-boiler and solvent-enriched product,

P3 is a high boiler enriched bottoms product comprising toluene-diisocyanat and

P4 is a toluenediisocyanate product stream lean in low-boilers, high-boilers and reaction residues.

10

3. The process of claims 1 or 2, wherein the product fraction P1 comprises 20 – 99% by weight of solvent, low-boilers and toluylenediisocyanate.

4. The process of any of claims 1 to 3, wherein the product fraction P2 comprises solvent, low boilers and toluylenediisocyanate.

15

5. The process of any of claims 1 to 4, wherein the product fraction P3 comprises toluenediisocyanate and 0.5-15% by weight of high-boilers.

20

6. The process of any of claims 1 to 5, wherein the product fraction P4 has a toluenediisocyanate concentration of at least 99.5% by weight and comprises less than 200 ppm by weight of solvent and / or chlorinated aromatic hydrocarbons, less than 100 ppm by weight hydrolyzable chlorine and less than 40 ppm by weight acidity.

25

Process for the purification of toluenediisocyanate incorporating a dividing-wall distillation column for the final purification

EPO - Munich
69

22. Okt. 2002

Summary:

The invention is directed to a process for the purification of toluenediisocyanate from a crude distillation feed comprising less than 2% by weight of phosgene by

- a) fractionating the crude distillation feed comprising less than 2% by weight of phosgene to remove the solvent and optionally the reaction residues to produce a crude toluylenediisocyanate feed comprising less than 20% by weight of solvent and
- b) separating the crude toluylenediisocyanat feed comprising less than 20% by weight of solvent in a dividing-wall distillation column into four product fractions P1 – P4, whereby

P1 is a vapor phase low-boiler and solvent-enriched gas stream,

P2 is a low-boiler and solvent-enriched product,

P3 is a high boiler enriched bottoms product comprising toluenediisocyanat and

P4 is a toluenediisocyanate product stream lean in low-boilers, high-boilers and reaction residues.



Fig. 1

